Research Article

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Infrared spectroscopy for ageing assessment of insulating oils *via* dielectric loss factor and interfacial tension

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Abstract: Along with the increase in demand for electricity, dependability requirements for the power supply system also increase. Transformers are the backbone of the electrical system; thus, they must function reliably and safely. In this study, a method based on Fourier-transform infrared spectroscopy technology is proposed for assessing the ageing level of insulating oils. Copper-catalysed thermal ageing is applied to four distinct insulating oils at a temperature of 120°C. The insulating oil ageing peaks are promptly measured using infrared spectroscopy, and the peak area ratios are associated with the interfacial tension and dielectric loss factor. It is observed that the peak area ratio of the ageing peaks and interfacial tension are significantly correlated, and this relationship can be used to determine the ageing condition of the insulating oil. The findings of this study can be used to design sensors for online monitoring of insulating oil quality.

Keywords: FT-IR, correlation analysis, insulating oils, interfacial tension, dielectric loss

1 Introduction

As the power industry has expanded, it has become more crucial than ever to transmit power supply systems safely. The transformer is the key component of the electrical power system, which serves as the foundation of the power system and defines the stability of the system. It is also one of the most expensive and accident-prone equipment of the power system. However, electrical and thermal strains that are present continuously during a transformer's operation on the insulating system [1] will raise transformer's safety risk and result in irreversible harm. Online monitoring of the insulation system has consequently become a popular area of study for academics. The reliability of the insulation of the transformer is related to the reliable operation of the power transformer. According to Zhou *et al.* [2], the safety and service life of the transformer are directly correlated with the operational quality and ageing of the insulating oil. Barkas *et al.* also mention that as the transformer insulation ages, the transformer itself also ages [3]. Therefore, evaluating the insulating oil's state is quite important. In particular, it indicates both the overall condition of the transformer and the oil quality of the transformer [4–7].

The extensive use of modern analytical techniques has made insulating oil testing simpler [8,9]. Infrared spectroscopy is one of the most frequently used detection strategies. The literature [10] compares popular techniques for determining the composition of structural families and has demonstrated that the infrared analysis detection approach is more accurate than conventional detection techniques. Infrared spectroscopy has the advantages of accurate detection, full functionality, low cost, and non-destructive properties [11]. These techniques have been used to study the physicochemical characteristics of insulating oils, the additive cation exchange capacity, and the ageing markers in mineral insulating fluids, lubricants, and oils [12–17].

Infrared spectroscopy has recently become widely used by academics to examine insulating oils and evaluate the condition of transformers. A technique to determine the amount of antioxidants present in insulating oils using near infrared (NIR) spectroscopy was proposed by Leong *et al.* [18]. Trnka *et al.* [19] studied the ageing process of insulating oils and described their thermal ageing degradation products using Fourier-transform infrared spectroscopy (FT-IR), a typical method for insulating oil testing

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[20,21] because NIR is an unconventional spectroscopic technique. By examining the relationship between the dielectric loss factor, interfacial tension, acid value, and the technical method for evaluating the ageing process of insulating oil based on FT-IR technology, Qian *et al.* [22] presented the early detection of transformer health and invisible problems. The peak area ratio of the infrared ageing characteristic peaks based on FT-IR technology was compared to the acid value of samples of insulating oil by Wang *et al.* [23]. However, it has not been extensively investigated how interfacial tension, dielectric loss factor, and the ageing process of insulating oils interact.

In order to create a sensor for real-time monitoring of insulating oil degradation, this article uses the FT-IR technique to investigate changes in the characteristic functional groups of ageing products and analyse the correlation between the ageing breakdown products (carbonyl, aldehyde, carboxyl, *etc.*) and the infrared spectra of those indicators (dielectric loss factor, interfacial tension, *etc.*).

2 Materials and methods

2.1 Main raw materials

For this test, four different types of insulating oil were selected: three of them are base oil No. 7, base oil No. 10, base oil No. 25, and the fourth is a finished oil with a minimum cold commissioning temperature of −10°C. In addition, antioxidant additive DBPC (2,6-di-tert-butyl-*p*-cresol, also known as T501), insulating paper, and copper wire with a diameter of 1 mm were also required.

2.2 Specimen preparation

The four aged insulating oils were vacuum-degassed, and the kraft insulating paper was dried and dehydrated. The four insulating oils were weighed separately, and each sample was 320 g. To prevent the influence of gas and moisture during the weighing process, the insulating oil samples were again vacuum-degassed and then placed in mill-mouth reagent bottles. The base oil No. 7 and base oil No. 10 were mixed with DBPC at 0.3% by mass and placed on an ultrasonic shaker for 3 h and kept at a constant temperature of 50°C.

Depending on the amount of insulating oil weighed, 40 g of copper wire was weighed and made into a spiral; the insulating paper was cut to 8 cm × 64 cm × 2 cm per copy and rolled into a cylindrical shape for fixing. The copper wire and insulating paper were vacuum-degassed and then placed in insulating oil. The mill-mouth reagent bottles were placed on a tray in an electric oven at 120°C to accelerate the ageing process, and the insulating oil samples were changed in the oven at regular intervals, the samples were taken every 7 days, and the ageing time was recorded (see Figure 1 for a flow chart of the test).

The colour and transparency of the insulating oil can visually reflect the quality of the oil. Traditionally, the colour of insulating oil is generally determined using the American Society for Testing and Materials (ASTM) D 1500 standard and is indicated by a colour index (CI) from a scale. Unaged insulating oil is clear and odourless, but as it ages, the transparency of the oil decreases and the smell becomes pungent [24]. The colour of the insulating oil changes from left to right as the ageing time increases, as shown in Figure 2.

2.3 Selection of ageing temperature

Yang *et al.* [25] found that the number of equipment with a transformer life of 11–15 years was more than twice that of

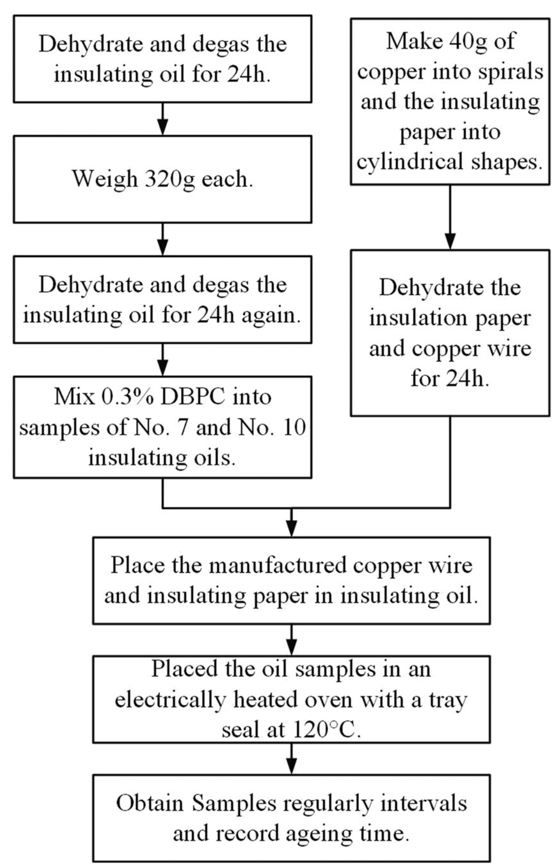

Figure 1: Test flow chart.

Figure 2: Ageing insulating oil colour change.

6–10 years, so it can be concluded that the life of general insulating oil is 11–15 years. According to Montsinger's law, the reduction in estimated life can be calculated by the 8° rule (an 8° increase in average life temperature over the maximum permissible operating temperature halves the estimated life of the oil insulation system [26]), so 120°C is chosen as the ageing temperature in this article.

2.4 Insulation oil performance index tests and methods

In order to measure and record the initial insulating oil sample family composition as well as the DBPC content of the insulating oil, the initial samples of the four different types of insulating oil were collected. The insulating oil was then scanned in the infrared, and the infrared spectra of the infrared liquid pools of 0.1 and 1 mm thickness were recorded, which are shown in Table 1.

The dielectric loss factor and interfacial tension of the ageing oil were tested. The measurements were taken two times, and the average value was calculated. The measurement method reference standards are shown in Table 2. The parameters selected for measurement were referred to the literature and the standard Guo Biao/Tui Jian 7595-2007 [27] quality of insulating oil in operation.

2.5 Testing of the dielectric loss factor

Referring to international electrotechnical commission (IEC) 60247 [28] to the determination of the dielectric loss factor, the electrode cups and electrodes were first cleaned with petroleum ether and later with deionised water. Then, they were dried and assembled the inside of the electrode cup is washed twice with a sample of the insulating oil to be tested and filled with about 50 mL of the test oil. A customised mode that allows the simultaneous determination of dielectric loss and volume resistivity was selected. The indicator can then be measured at 90°C.

Table 1: Initial insulating oil sample test parameters

Insulating oil sample markings	Insulating oil sample composition	Content in insulating oil (%)
No. 1	Cycloalkyl	0.32
No. 2	Cycloalkyl	0.32
No. 2	Cycloalkyl	0.27
No. 2	Non-cycloalkyl	0.02

2.6 Testing of interfacial tension

Referring to international organization for standardization (ISO) 6295 [29] to the determination of the interfacial tension, the platinum ring and the sample cup were kept clean. The sample cup was filled with deionised water to the middle scale, and the tension value of the water to be tested was selected. Then, the lifting table was raised until the platinum ring was submerged in the sample cup by 5–6 mm. Using the burette, a sample of oil was taken and the oil was dropped along the wall of the cup until the height of the sample reached the top mark of the cup. Finally, the sample was selected to test the water–oil interfacial tension, and the data were recorded.

2.7 Infrared test methods

The infrared test analysis was carried out in a removable liquid cell (Model: HARRICK HPL-C-13) with a potassium bromide window sheet +1 mm, measuring the infrared wavelength range from 4,000 to 600 cm^{-1}. The advantage of the removable liquid cell is that it is easy to clean, but the disadvantage is that the layer thickness varies slightly for each test depending on the loading and unloading effort. According to Lambert's law, the absorbance is proportional to the layer thickness of the liquid cell. Therefore, it is necessary to always check and verify the optical path length after cleaning the removable liquid cell. In order to determine the peak areas, the ratio of the aged peak's peak area to the reference peak's peak area was calculated, as stated in ASTM E1252 [30], "Infrared Spectroscopy of the Structural Group Composition of Mineral Insulating

Table 2: Measurement reference standards

Parameter name	Reference standards
Dielectric loss factor	IEC 60247 [28] measurement of dielectric loss factor of liquid insulating materials
Interfacial tension	ISO 6295 [29] determination of oil-to-water interfacial tension of petroleum products (circular method)

Oils and Lubricants." The infrared characteristic peaks appearing during the ageing process were explored, and the peak areas of the characteristic peaks were recorded to find the correlation between the dielectric loss factor and interfacial tension and the intensity of the characteristic peaks.

2.8 Correlation analysis

For the aged oil samples, the dielectric loss factor and interfacial tension were tested and recorded as two conventional ageing indicators. The infrared full spectra of all new and aged insulating oils were recorded to find the characteristic peaks and their corresponding functional groups during the ageing process. The correlation coefficients between the peak area ratios of the ageing peaks and the conventional ageing indicators were calculated and fitted to the regression curves. The correlation between the peak area ratio of each ageing peak and the conventional ageing index was analysed. As this study was only designed to find the correlation between the two variables, it is sufficient to express with the Pearson correlation coefficient [31], and no further regression analysis was required for variable relationships with Pearson correlation coefficients ≥0.005. In order to obtain the conventional ageing index of the insulating oil samples from the IR spectra, the "peak area ratio" was set as the independent variable and the conventional ageing index was set as the dependent variable in the regression equation.

3 Results

3.1 FT-IR results for base oil No. 1

The FT-IR method in this article does not assess the ageing state directly on the basis of colour. Therefore, the study in this article is based on a baseline correction. Determination of the ageing degree of the insulating oil is based on the ageing colour and transparency of the insulating oil; a total of 21 ageing samples with different ageing degrees were taken for oil No. 1. The ageing times were 168, 336, 504, 672, 864, 1,008, 1,176, 1,344, 1,512, and 1,680 h. The samples to be tested were injected into the liquid pool, and the scanning wave number range was set to 4,000–600 cm^{-1} in the software OPUS for infrared spectroscopy testing.

Figure 3 shows the infrared spectra of the ageing insulating oil sample of oil No. 1 in the 4,000–600 cm^{-1} wave number band. In Figure 3, the lower image is a local zoom in on the upper image in the range 1,800–1,600 cm^{-1}. The area of local enlargement is the area of focus in this study. This is also the case for Figures 4, 5, 6. It can also be found through Figure 3 that the 3,650 cm^{-1} spectral band is also sensitive to the ageing process, and Polansky *et al.* [32] studied the ageing process of insulating oils through this band. However, it can be found through Figure 3 that the 1,600–1,800 cm^{-1} spectral band is more sensitive to the ageing process and has been less studied. Therefore, the 3,650 cm^{-1} spectral band will not be investigated in this article.

The results show that the absorption peak signal is extremely strong in the 3,500–2,500 cm^{-1} wave number band, which is the vibration of the C–H bond and is the absorption peak of the hydrocarbon of the main body of the insulating oil. However, in the 1,500–800 cm^{-1} wave number band, the absorption peaks are more haphazard and have no specific pattern.

3.2 FT-IR results for base oil No. 2

A total of 19 ageing samples of oil No. 2 were taken. The ageing times were 168, 336, 504, 672, 864, 1,008, 1,176, 1,344, and 1,680 h, respectively. Infrared spectroscopy of the base oil No. 2 was carried out according to the method in Section 3.1.

Figure 4 shows the infrared spectra of the ageing insulating oil sample of oil No. 2 in the 4,000–600 cm^{-1} wave number band.

The results show that after ageing to 1,176 h, the insulating oil of No. 2 samples shows a gradual increase in absorption peak intensity with ageing time at 1,775, 1,697, 1,682, 1,660, 1,648, and 1,599 cm^{-1}.

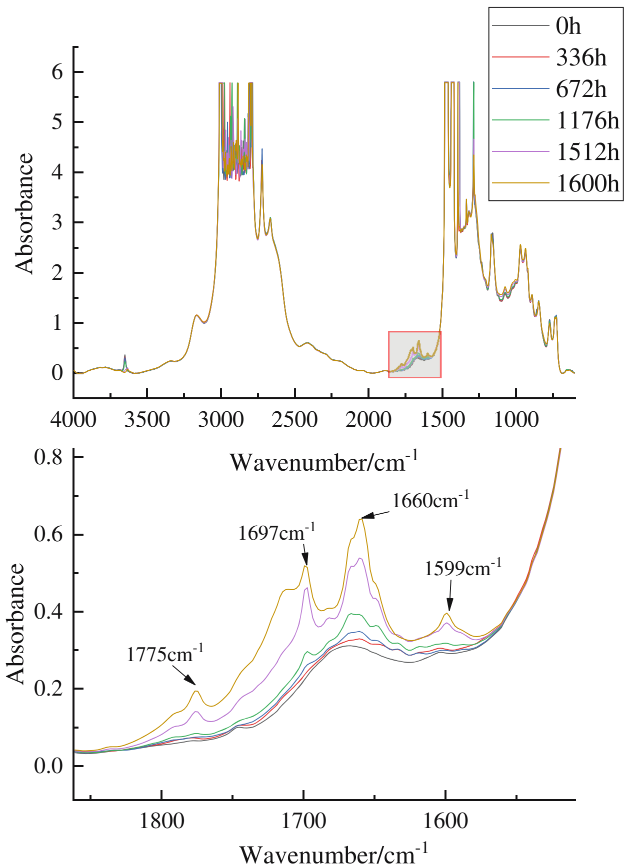

Figure 3: The upper image is infrared full spectrum of insulating oil No. 1, and the lower image is local magnification of the 1,600–1,800 cm^{-1} band.

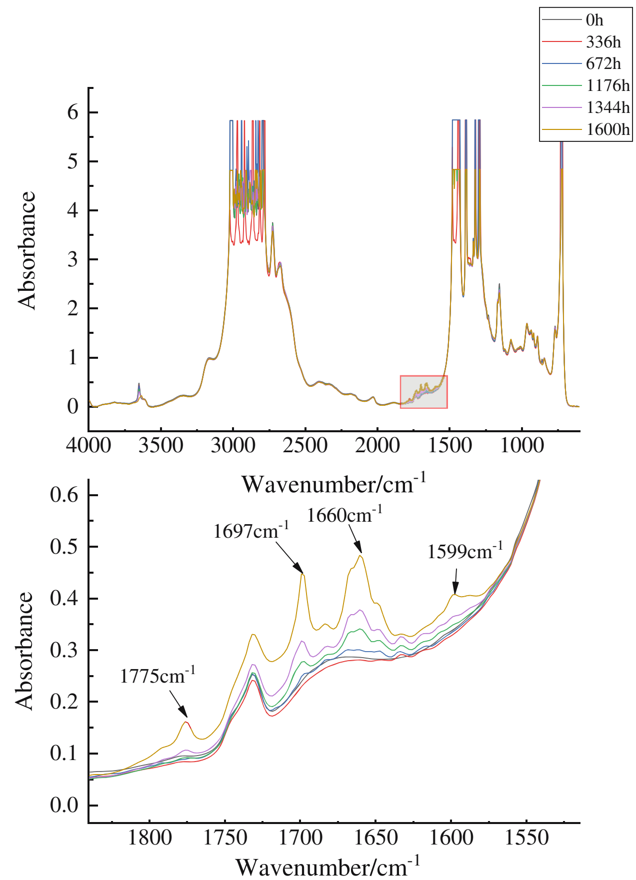

Figure 4: The upper image is infrared full spectrum of insulating oil No. 2, the lower image is local magnification of the 1,550–1,800 cm^{-1} band.

3.3 FT-IR results for base oil No. 3

A total of nine samples were aged for oil No. 3. The ageing times were 168, 242, 336, 700, 840, 1,176, and 1,344 h, respectively. Infrared spectroscopy of base oil No. 3 was also carried out according to the procedure in Section 3.1.

Figure 5 shows the infrared spectra of the ageing insulating oil sample of oil No. 3 in the 4,000–600 cm^{-1} wave number band.

According to the results shown in Figure 5, it is found that the infrared absorption peaks at 1,775, 1,697, 1,683, 1,660, 1,648, and 1,606 cm^{-1} show a gradual increase in peak height and absorption intensity with increasing ageing time, unlike insulating oil No. 1 and No. 2, with no clear infrared absorption peak at 1,599 cm^{-1}.

3.4 FT-IR results for base oil No. 4

A total of 15 samples were aged for insulating oil No. 4. The ageing times were 168, 336, 504, 840, 1,020, 1,080, and 1,250 h, respectively. Infrared spectroscopy of base insulating oil No. 4 was also carried out according to the method in Section 3.1.

Figure 6 shows the infrared spectra of the ageing insulating oil sample of oil No. 4 in the 4,000–600 cm^{-1} wave number band.

Comparing all the aforementioned data, it can be found that the ageing peaks of insulating oil No. 1, No. 2, and No. 3 with DBPC added are basically the same, with the ageing product peaks located at 1,775, 1,697, and 1,660 cm^{-1}. The infrared spectrum for the severely aged sample, shows a peak at 1,599 cm^{-1}. The ageing peaks for insulating oil No. 4 are located at 1,788, 1,720, and 1,644 cm^{-1}.

3.5 Results of correlation analysis

3.5.1 Selection of reference peaks

In order to reduce the error due to the influence of layer thickness, the absorption peak in the infrared spectrum of the aged insulating oil samples, which basically did

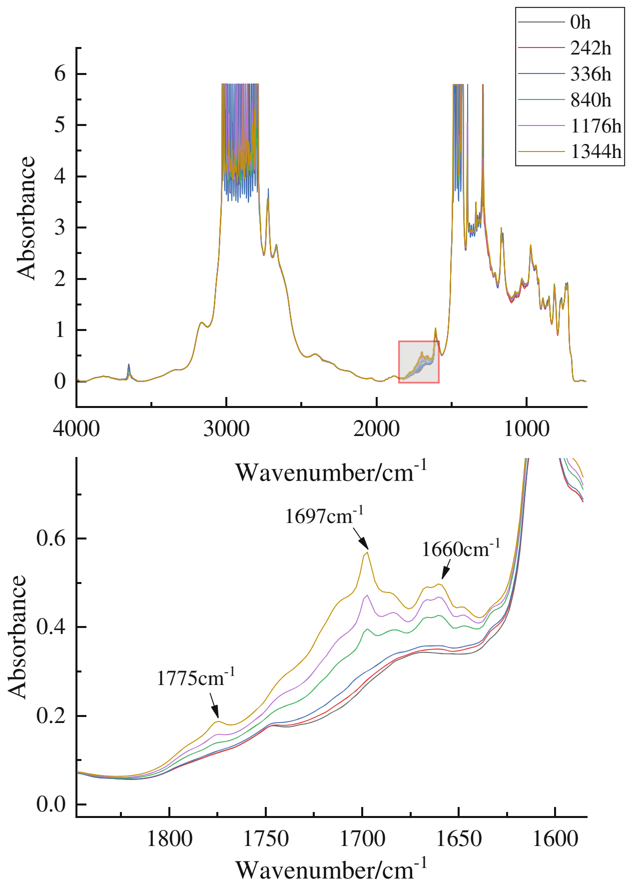

Figure 5: The upper image is infrared full spectrum of insulating oil No. 3, the lower image is local magnification of the 1,600–1,800 cm^{-1} band.

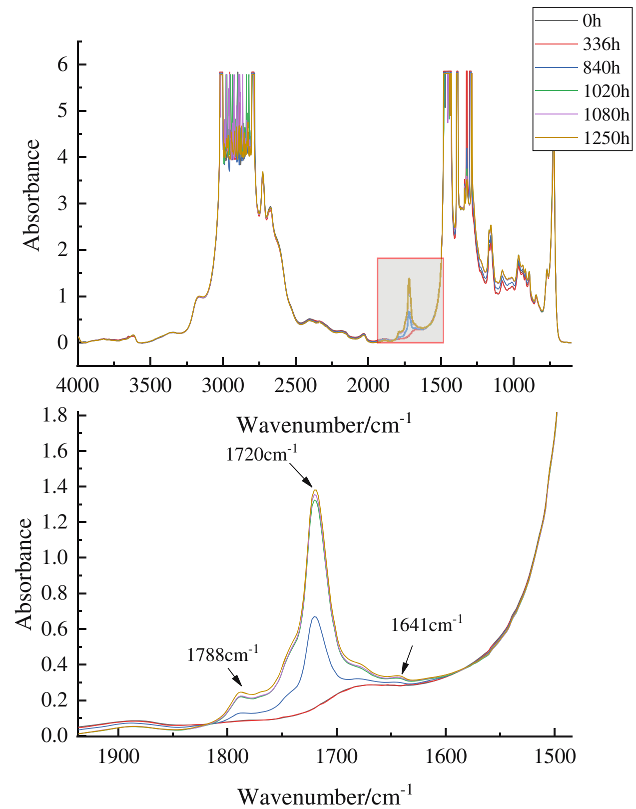

Figure 6: The upper image is infrared full spectrum of insulating oil No. 4, the lower image is local magnification of the 1,500–1,900 cm^{-1} band.

not change with ageing time, is chosen as the reference peak in the correlation analysis. The infrared absorption peak at 3,165 cm^{-1} was discovered by analysing the infrared spectra in Sections 3.1–3.4. As ageing time increased, Figure 7 shows that the peak area and peak height at 3,165 cm^{-1} remained almost unchanged. Therefore, the infrared absorption peak at 3,165 cm^{-1} is selected as the reference peak in this article.

3.5.2 Base oil No. 1 correlation analysis results

There were 19 samples of aged insulating oil. Two samples of unaged insulating oil were used: one without DBPC and the other containing 0.3% DBPC. There were 21 samples of insulating oil in total. To conduct a correlation analysis, the data were loaded into SPSS program. The dependent variables Y are the dielectric loss factor and interfacial tension, while the independent variable X is the ratio of the peak area of the ageing peak to the peak area of the reference peak at 3,165 cm^{-1}. The dielectric loss factor and interfacial tension were regressed on the peak area ratios of 1,775/3,165, 1,697/3,165, 1,660/3,165, 1,599/3,165, and 1,599/3,165 cm^{-1}, respectively, using a polynomial regression of orders 1, 2, and 3. Tables 3 and 4 display the findings of the goodness-of-fit and significance tests.

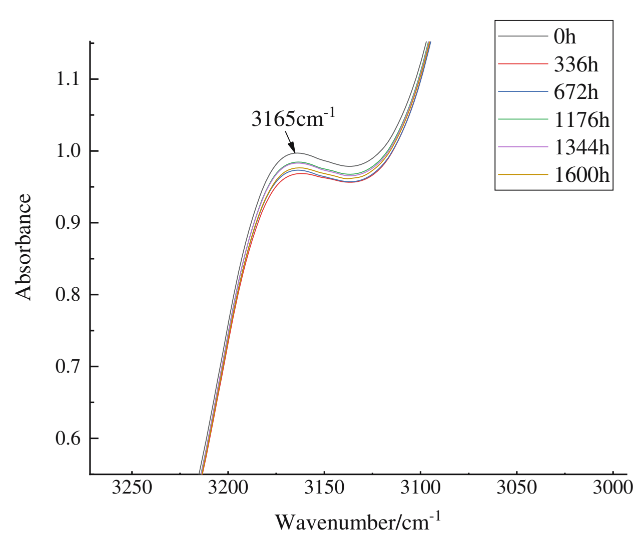

Figure 7: Absorption peak at 3,165 cm^{-1}.

Table 3: Fitting results for the dielectric loss factor of base oil No. 1

	Parameters	Linear equation	Equation of the two degree	Equation of the third degree
Dielectric loss factor against	Significance	0	0	0.001
1,775/3,165 cm^{-1} peak area ratio	R^2	0.516	0.594	0.597
Dielectric loss factor against	Significance	0	0.002	0.006
1,697/3,165 cm^{-1} peak area ratio	R^2	0.508	0.509	0.509
Dielectric loss factor against	Significance	0	0.001	0.002
1,660/3,165 cm^{-1} peak area ratio	R^2	0.559	0.566	0.580
Dielectric loss factor against	Significance	0.001	0.002	0.007
1,599/3,165 cm^{-1} peak area ratio	R^2	0.457	0.497	0.502

The fitting curve of the dielectric loss factor and interfacial tension against the peak area ratios of 1,775/3,165, 1,697/3,165, 1,660/3,165, and 1,599/3,165 cm^{-1} are plotted, respectively, as shown in Figures 8 and 9.

The aforementioned data show that the goodness-of-fit of the fitting curves for the dielectric loss factor of oil No. 1 and the peak area ratios of the peaks at 1,775, 1,697, 1,660, and 1,599 cm^{-1} are all less than 0.6. The best model for the interfacial tension against peak area ratio of 1,660/3,165 cm^{-1} was a cubic model with a goodness-of-fit of 0.912. There is no correlation above 0.9 between the interfacial tension and the peak area ratios of 1,775/3,165, 1,697/3,165, and 1,599/3,165 cm^{-1}.

3.5.3 Base oil No. 2 correlation analysis results

There were 17 samples of ageing insulating oil and 2 samples of unaged insulating oil with and without 0.3% DBPC added, respectively. There were 19 samples of insulating oil in total. The analysis method was the same as that used in Section of 3.4.2. The results are shown in Tables 5 and 6.

The fitting curve of the dielectric loss factor and interfacial tension against the peak area ratios of 1,775/3,165, 1,697/3,165, 1,660/3,165, 1648/3,165, and 1,599/3,165 cm^{-1} are plotted, respectively, as shown in Figures 10 and 11.

The best model for each group is as follows: the best goodness-of-fit of the fitted curve for the dielectric loss factor and the peak area ratio of 1,697/3,165 and 1,660/3,165 cm^{-1} is about 0.8, higher than the peak area ratio of the 1,775/3,165 and 1,599/3,165 cm^{-1}.

However, the goodness-of-fit of the optimal models for the interfacial tension to 1,775/3,165 and 1,697/3,165 cm^{-1} peak area ratios is only 0.607 and 0.641, respectively.

3.5.4 Base oil No. 3 correlation analysis results

There were eight samples of ageing insulating oil and one sample of unaged insulating oil. The total number of insulating oil samples was 9. The method is the same as in Section of 3.4.2. The results of goodness-of-fit and significance are shown in Table 7.

It is shown that the best goodness-of-fit for the interfacial tension to 1,697/3,165 cm^{-1} peak area ratio is 0.769; the best goodness-of-fit for the interfacial tension to 1,660/3,165 cm^{-1} peak area ratio is 0.773 (Figure 12).

3.5.5 Base oil No. 4 correlation analysis results

There were 14 samples of aged insulating oil and 1 sample of unaged insulating oil. The total number of insulating oil

Table 4: Fitting results for the interfacial tension of base oil No. 1

	Parameters	Linear equation	Equation of the two degree	Equation of the third degree
Interfacial tension against	Significance	0	0	0
1,775/3,165 cm^{-1} peak area ratio	R^2	0.744	0.819	0.861
Interfacial tension against	Significance	0	0	0
1,697/3,165 cm^{-1} peak area ratio	R^2	0.738	0.762	0.764
Interfacial tension against	Significance	0	0	0
1,660/3,165 cm^{-1} peak area ratio	R^2	0.855	0.863	0.912
Interfacial tension against	Significance	0	0	0
1,599/3,165 cm^{-1} peak area ratio	R^2	0.724	0.763	0.780

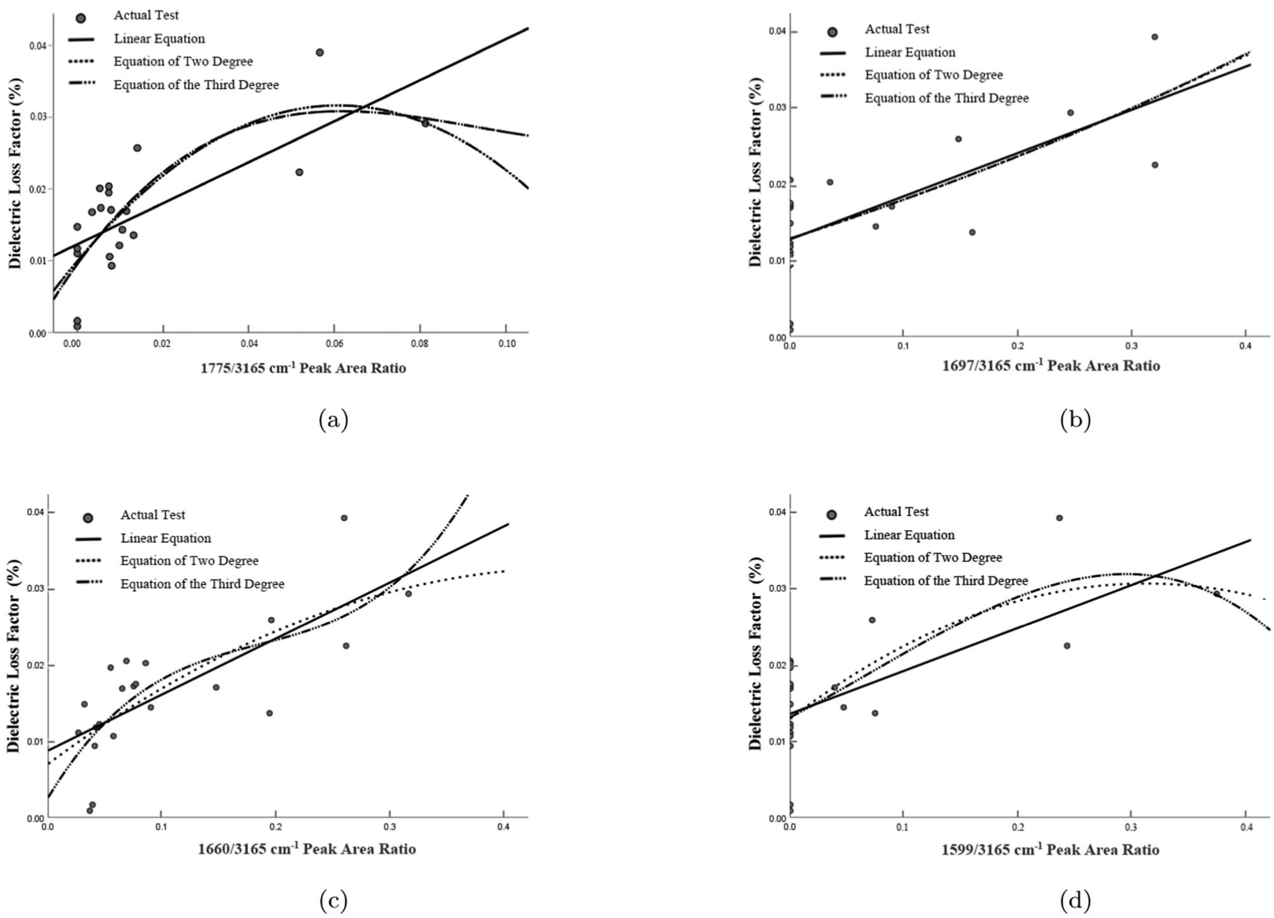

Figure 8: Fitting curve for dielectric loss factor against peak area ratio of base insulating oil No. 1: (a) fitting curve graph of dielectric loss factor against 1,775/3,165 cm^{-1} peak area ratio, (b) fitting curve graph of dielectric loss factor against 1,697/3,165 cm^{-1} peak area ratio, (c) fitting curve graph of dielectric loss factor against 1,660/3,165 cm^{-1} peak area ratio, and (d) fitting curve graph of dielectric loss factor against 1,599/3,165 cm^{-1} peak area ratio.

samples of insulating oil No. 4 was 15. The method is the same as Section of 3.4.2, and the results are shown in Tables 8 and 9.

The fitting curve of the dielectric loss factor and interfacial tension against the peak area ratios of 1,788/3,165, 1,720/3,165, and 1,644/3,165 cm^{-1} is plotted respectively, as shown in Figures 13 and 14. The results show a better fit for this group.

4 Discussion

4.1 Analysis of infrared spectral results

Calemma *et al.* [33] and Castro *et al.* [34] showed that the stretching vibrations of the carbonyl groups of ketones, acids, lipids, and aldehydes are all located between 1,850–1,660 cm^{-1}, while the C=O absorption peak of carboxylic acids is located near 1,700 cm^{-1}. Lievens *et al.* [35] found that the C=O absorption frequencies of aldehydes and ketones are close to each other and the C=O absorption peak position of aldehydes is usually 10–15 cm^{-1} larger than that of ketones, and the functional groups with absorption frequencies at 1,600 cm^{-1} are C=C and $-NH_2$ in the aromatic ring. Combining the data from the previous section, it can be concluded that the ageing peak at 1,697 cm^{-1} is probably the C=O stretching vibration of a carboxylic acid and the ageing absorption peaks at 1,667 and 1,660 cm^{-1} are probably the C=O stretching vibration peaks of aldehydes and ketones. The absorption peaks of nitrogen oxides of lubricating oils mentioned in the standard [36] are located around 1,650–1,610 cm^{-1}, so the ageing peak at 1,599 cm^{-1} may be the absorption peak of

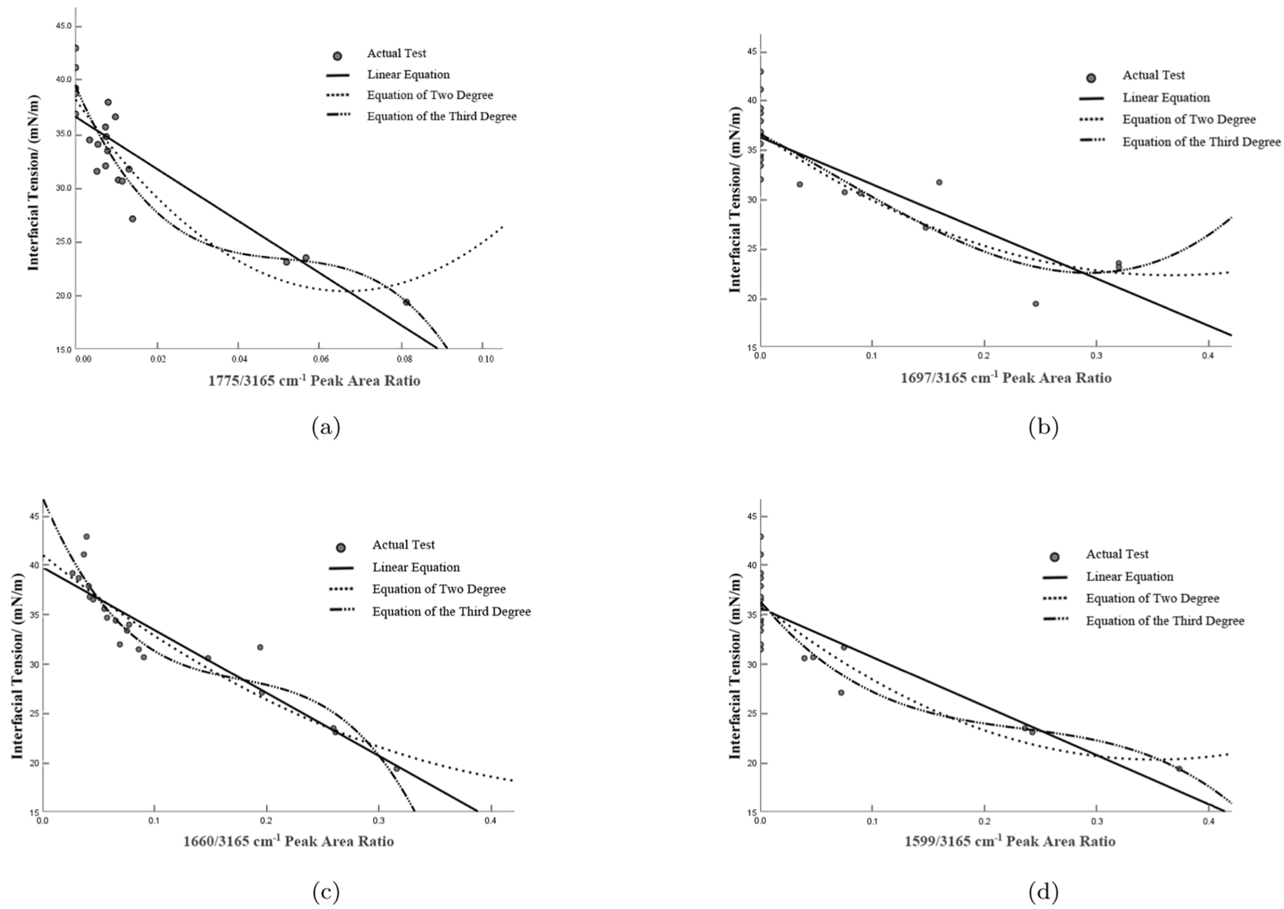

Figure 9: Fitting curve for interfacial tension against peak area ratio of base insulating oil No. 1: (a) Fitting curve graph of interfacial tension against 1,775/3,165 cm^{-1} peak area ratio, (b) fitting curve graph of interfacial tension against 1,697/3,165 cm^{-1} peak area ratio, (c) fitting curve graph of interfacial tension against 1,660/3,165 cm^{-1} peak area ratio, and (d) fitting curve graph of interfacial tension against 1,599/3,165 cm^{-1} peak area ratio.

Table 5: Fitting results for the dielectric loss factor of base oil No. 2

	Parameters	Linear equation	Equation of two degree	Equation of the third degree
Dielectric loss factor against	Significance	0	0.001	0.002
1,775/3,165 cm^{-1} peak area ratio	R^2	0.550	0.557	0.627
Dielectric loss factor against	Significance	0	0	0
1,697/3,165 cm^{-1} peak area ratio	R^2	0.658	0.663	0.819
Dielectric loss factor against	Significance	0	0	0
1,660/3,165 cm^{-1} peak area ratio	R^2	0.697	0.703	0.837
Dielectric loss factor against	Significance	0.002	0.002	0.002
1,599/3,165 cm^{-1} peak area ratio	R^2	0.456	0.456	0.456

Table 6: Fitting results for the dielectric loss factor of base oil No. 2

	Parameters	Linear equation	Equation of two degree	Equation of the third degree
Interfacial tension against	Significance	0.002	0.001	0.002
1,775/3,165 cm^{-1} peak area ratio	R^2	0.425	0.607	0.607
Interfacial tension against	Significance	0	0	0.001
1,697/3,165 cm^{-1} peak area ratio	R^2	0.553	0.641	0.641
Interfacial tension against	Significance	0	0	0
1,660/3,165 cm^{-1} peak area ratio	R^2	0.649	0.782	0.832

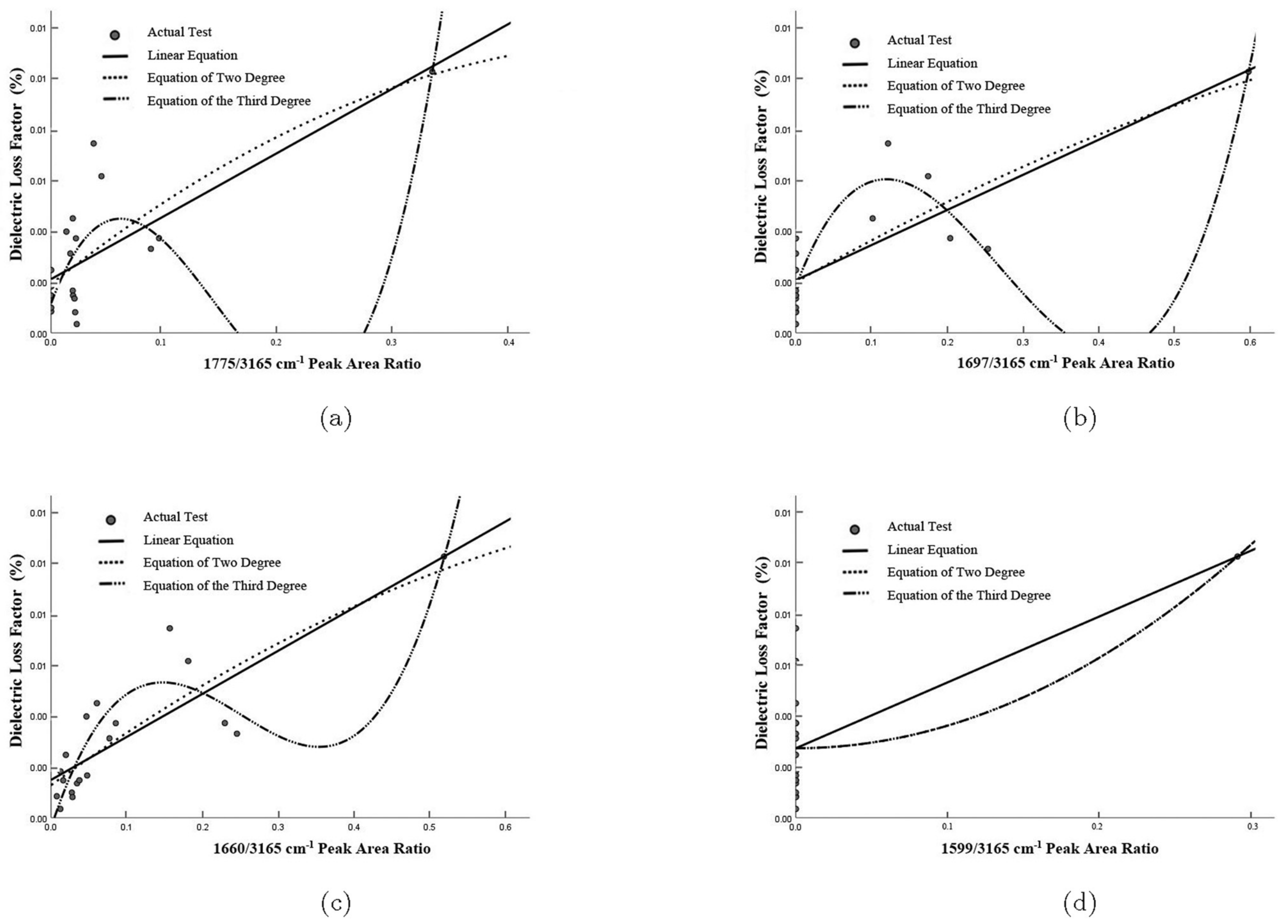

Figure 10: Fitting curve for dielectric loss factor against peak area ratio of base insulating oil No. 2: (a) fitting curve graph of dielectric loss factor against 1,775/3,165 cm^{-1} peak area ratio, (b) fitting curve graph of dielectric loss factor against 1,697/3,165 cm^{-1} peak area ratio, (c) fitting curve graph of dielectric loss factor against 1,660/3,165 cm^{-1} peak area ratio, and (d) fitting curve graph of dielectric loss factor against 1,599/3,165 cm^{-1} peak area ratio.

$-NH_2$ or nitrogen oxides of ageing products; as the C═O of esters appears at 1,750–1,725 cm^{-1}, so it can be analysed that the ageing process of insulating oil does not produce a large amount of esters.

4.2 Analysis of correlation results

The peak area ratio of the ageing peak to the reference peak, together with the dielectric loss factor and interfacial

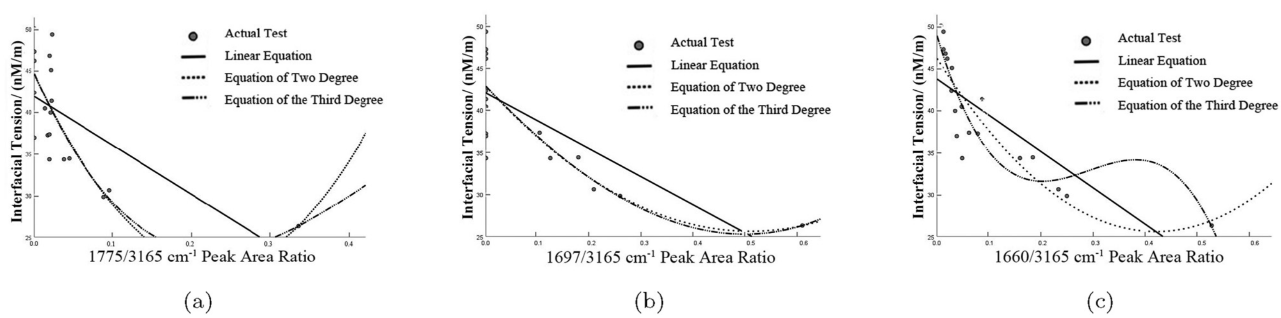

Figure 11: Fitting curve for interfacial tension against peak area ratio of base insulating oil No. 2: (a) fitting curve graph of interfacial tension against 1,775/3,165 cm^{-1} peak area ratio, (b) fitting curve graph of interfacial tension against 1,697/3,165 cm^{-1} peak area ratio, and (c) fitting curve graph of interfacial tension against 1,660/3,165 cm^{-1} peak area ratio.

Table 7: Fitting results for the interfacial tension of base oil No. 3

	Parameters	Linear equation	Equation of two degree	Equation of the third degree
Interfacial tension against	Significance	0.003	0.014	0.048
1,775/3,165 cm^{-1} peak area ratio	R^2	0.733	0.759	0.769
Interfacial tension against	Significance	0.002	0.012	0.046
1,697/3,165 cm^{-1} peak area ratio	R^2	0.770	0.772	0.773

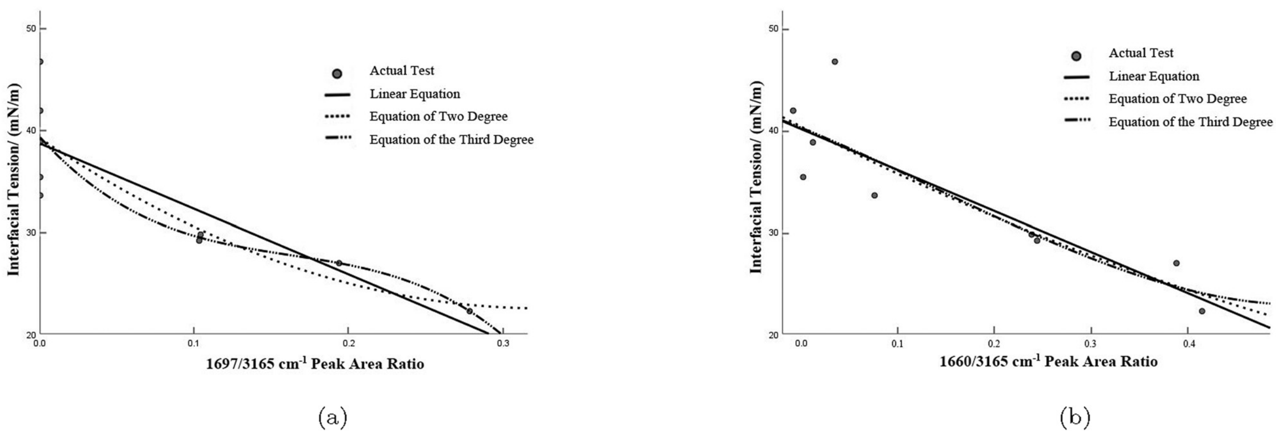

Figure 12: Fitting curve for interfacial tension against peak area ratio of base insulating oil No. 3 (a) Fitting curve graph of interfacial tension against 1,697/3,165 cm^{-1} peak area ratio and (b) fitting curve graph of interfacial tension against 1,660/3,165 cm^{-1} peak area ratio.

Table 8: Fitting results for the dielectric loss factor of base oil No. 4

	Parameters	Linear equation	Equation of two degree	Equation of the third degree
Dielectric loss factor against	Significance	0	0	0
1,775/3,165 cm^{-1} peak area ratio	R^2	0.740	0.876	0.883
Dielectric loss factor against	Significance	0	0	0
1,697/3,165 cm^{-1} peak area ratio	R^2	0.691	0.769	0.856
Dielectric loss factor against	Significance	0	0	0
1,660/3,165 cm^{-1} peak area ratio	R^2	0.763	0.882	0.964

Table 9: Fitting results for the interfacial tension of base oil No. 4

	Parameters	Linear equation	Equation of the two degree	Equation of the third degree
Interfacial tension against	Significance	0	0	0
1,775/3,165 cm^{-1} peak area ratio	R^2	0.787	0.886	0.942
Interfacial tension against	Significance	0	0	0
1,697/3,165 cm^{-1} peak area ratio	R^2	0.864	0.933	0.943
Interfacial tension against	Significance	0	0	0
1,660/3,165 cm^{-1} peak area ratio	R^2	0.819	0.914	0.940

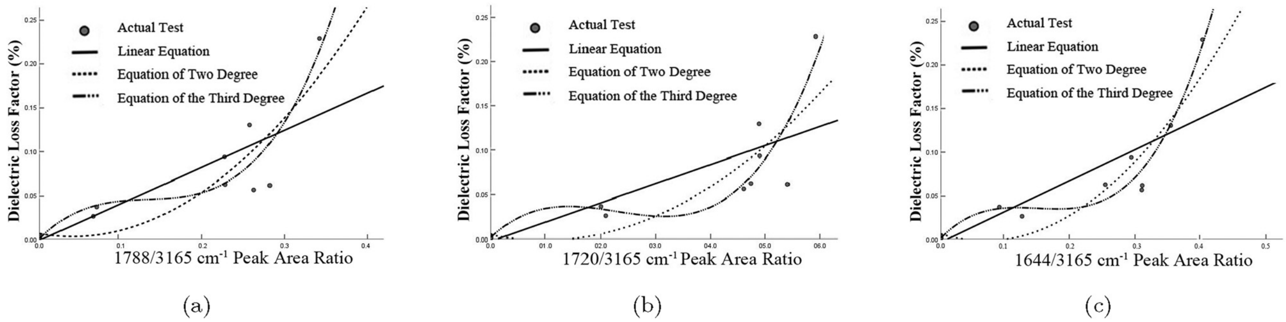

Figure 13: Fitting curve for dielectric loss factor against peak area ratio of base insulating oil No. 4 (a) fitting curve graph of dielectric loss factor against 1,788/3,165 cm^{-1} peak area ratio, (b) fitting curve graph of dielectric loss factor against 1,720/3,165 cm^{-1} peak area ratio, and (c) fitting curve graph of dielectric loss factor against 1,644/3,165 cm^{-1} peak area ratio.

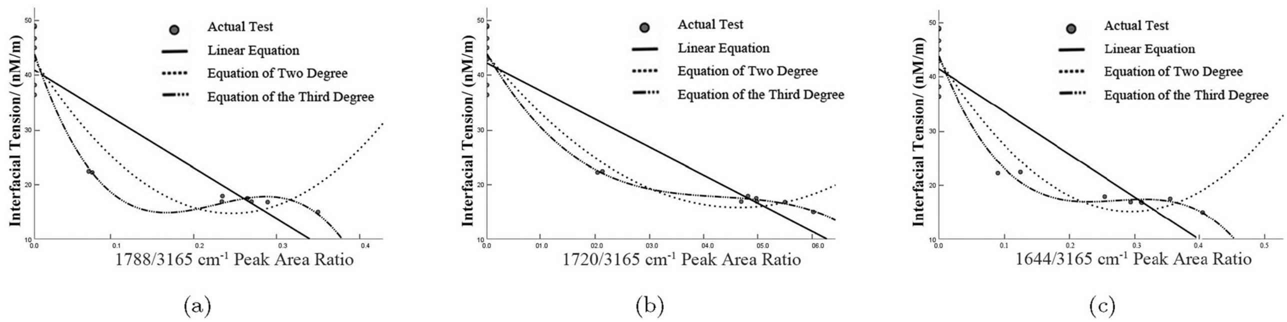

Figure 14: Fitting curve for interfacial tension against peak area ratio of base insulating oil No. 4 (a) fitting curve graph of interfacial tension against 1,788/3,165 cm^{-1} peak area ratio, (b) fitting curve graph of interfacial tension against 1,720/3,165 cm^{-1} peak area ratio, and (c) fitting curve graph of interfacial tension against 1,644/3,165 cm^{-1} peak area ratio.

tension of the insulating oil sample, was investigated, and the following results were discovered:

- The correlation between the dielectric loss factor and the peak area ratios of the 1,775 cm^{-1}, 1,697 cm^{-1}, 1,660 cm^{-1}, and 1,599 cm^{-1} peaks for oil No. 1 is not high, and the goodness-of-fit is less than 0.6. It is not possible to establish a basis for evaluating the level of ageing in insulating oil samples. Although the correlation between the interfacial tension and the peak area ratios of the 1,775, 1,697, and 1,599 cm^{-1} peaks did not reach above 0.9, it is still possible to establish a basis for determining the level of ageing of the insulating oil samples because the interfacial tension and the peak area ratios of the 1,660 cm^{-1} peaks have a strong correlation.
- The correlation between the dielectric loss factor and the peak area ratio of the 1,697 and 1,660 cm^{-1} peaks for insulating oil No. 2 is greater than 0.8, which can be used as a criterion for the ageing of insulating oil samples to a certain extent. Simultaneously, the correlation between the interfacial tension and the peak area ratio of the ageing peaks at 1,775 and 1,697 cm^{-1} is not high, and the goodness-of-fit is low, which cannot reflect the ageing of the insulating oil samples.
- The goodness-of-fit values between the peak area ratio of the 1,697 and 1,660 cm^{-1} peaks and the interfacial tension of insulating oil No. 3 are both greater than 0.75., although the correlation did not reach above 0.9, but still it can reflect the degree of ageing of the insulating oil samples to a certain extent.
- The correlation between the dielectric loss factor of insulating oil No. 4 and the peak area ratio of 1,644/3,165 cm^{-1} is 0.964, which is a strong correlation. Simultaneously, the correlation between the interfacial tension and the peak area ratios of 1,778, 1,720, and 16,444 cm^{-1} peaks are all strong and the goodness-of-fit is greater than 0.940. It can be seen that the dielectric loss factor and interfacial tension are good indicators of the degree of ageing of an insulating oil sample and can be used as a basis for determining the degree of ageing of an insulating oil sample.

After correlation analysis, regression equations with a high partial correlation are obtained, offering the possibility of IR detection of the interfacial tension of the insulating oil.

5 Conclusion

In summary, the following conclusions can be drawn from this study.

- In terms of ageing time order, the characteristic peaks of the four insulating oil samples are 1,660, 1,697, 1,775, and 1,599 cm^{-1}, for which the 1,599 cm^{-1} ageing peak only appeared after severe ageing of the insulating oil samples.
- The ageing peak of carboxylic acids (C═O) stretching vibration is appeared at 1,697 cm^{-1}, and the C═O stretching vibration peak for aldehydes and ketones is appeared at 1,660 and 1,667 cm^{-1}; the ageing peak at 1,599 cm^{-1} is the absorption peak for N–H in the ageing products or the absorption peak for nitrogen oxides; no significant esters are produced during the ageing of the oil.
- The correlation between the dielectric loss factors of the four insulating oils and the ageing absorption peaks in the IR wavelength range 1,700 to 1,660 cm^{-1} is not strong, and the goodness-of-fit of the one-dimensional cubic fitting curve is basically less than 0.9. However, the correlation between the interfacial tensions of the four insulating oils and the ageing absorption peaks in the IR wavelength range 1,700 to 1,660 cm^{-1} is strong, and the goodness-of-fit of the one-dimensional cubic fit curve is basically greater than 0.8. Therefore, the interfacial tension of the oils can be determined to some extent by testing the peak area ratio of the ageing peaks by FT-IR.

In this article, an innovative method for the evaluation of insulating oil age and latent transformer faults is established by *in situ* FT-IR. At the same time, a sensor for real-time tracking of the insulation oil age process is developed with much higher accuracy. At the same time, the relationship between the degree of ageing of the insulating oil and the dielectric loss factor and interfacial tension is investigated in greater depth. This method can quickly test the ageing degree of insulating oil and provide a feasible way for online monitoring of transformers and can effectively improve the safety and reliability of transformer operation.

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References

[1] Pradhan MK, Ramu TS. On the estimation of elapsed life of oil-immersed power transformers. IEEE Trans Power Delivery. 2005;20:1962–9.
[2] Zhou C, Jiang S, Yin J, Cai S, Chen Y. Study on the change of acid value of transformer oil during thermal ageing. Insulation Materials. 2015;48:46–48+54.
[3] Barkas DA, Chronis I, Psomopoulos C. Failure mapping and critical measurements for the operating condition assessment of power transformers. Energy Reports. 2022;8:527–47.
[4] Alshehawy AM, Mansour D-EA, Ghali M, Rezk A. Evaluating the impact of ageing in field transformer oil using optical spectroscopy techniques. In: Proceedings of the IEEE 19th International Conference on Dielectric Liquids (ICDL), Manchester, England, 2017 Jun 25–29. 2017.
[5] Kohtoh M, Kaneko S, Okabe S, Amimoto T. Aging effect on electrical characteristics of insulating oil in field transformer. IEEE Trans Dielectrics Electr Insulation. 2009;16:1698–706.
[6] Zhang BQ, Ma YL, Guan R, Bai ST, Li J, Hu WT. Performance optimization of ANN method for oil chromatography fault diagnosis of oil-filled electrical equipment. Guangdong Electric Power, 2021;34(6):39–47.
[7] Baird PJ, Herman H, Stevens GC, Jarman PN. Non-destructive measurement of the degradation of transformer insulating paper. IEEE Trans Dielectrics Electr Insulation. 2006;13:309–18.
[8] Bustamante S, Manana M, Arroyo A, Castro P, Laso A, Martinez R. Dissolved gas analysis equipment for online monitoring of transformer oil: a review. Sensors. 2019;19:4057.
[9] Li J, Zhang Q, Wang K, Wang J, Zhou T, Zhang Y. Optimal dissolved gas ratios selected by genetic algorithm for power transformer fault diagnosis based on support vector machine. IEEE Trans Dielectrics Electr Insulation. 2016;23:1198–206.
[10] Feng L, Meng Y. Experimental study of DL/T 929-2005 “Infrared spectroscopic determination of structural family composition of insulating oils and lubricating oils”. Power Standardization Technol Economy. 2006;1:8–11.
[11] Zhang X, Wang J, Gao Y, Jiao L. Design of a miniature infrared spectrum detection system based on quantum cascade laser. In: Proceedings of the International Conference on Optoelectronic Materials and Devices (ICOMD), Guangzhou, Peoples R China, 2022 Dec 10–12, 2021.
[12] Hadjadj Y, Fofana I, Jalbert J, Insulating oil decaying assessment by FTIR and UV-Vis spectrophotometry measurements. In: Proceedings of the IEEE Annual Report Conference on Electrical Insulation and Dielectric Phenomena (CEIDP), Shenzhen, Peoples R China; 2013 Oct 20–23, 2013. p. 1310–3.
[13] Winterfield C, van de Voort FR. Automated acid and base number determination of mineral-based lubricants by Fourier transform infrared spectroscopy: commercial laboratory evaluation. Jala. 2014;19:577–86.

[14] Meng X, Li L, Ye Q, van de Voort F. Fourier transform infrared (FTIR) spectroscopy as a utilitarian tool for the routine determination of acidity in ester-based oils. J Agricult Food Chem. 2015;63:8333–8.
[15] Zhang J, Ke Y, Ma X, Ma R, He J, Zhang X. Application of modern analytical techniques in transformer oil performance testing. Insulation Materials. 2017;50:15–21.
[16] Hironmay D, Javin N, MunMoonSun N, Superdao J, Dipankar S. Transformer oil quality diagnostic using spectroscopy techniques - a review. ADBU J Electr Electron Eng. 2019;3:43–50.
[17] Zhu X, Zhong C, Zhe J. Lubricating oil conditioning sensors for online machine health monitoring - A review. Tribol Int. 2017;109:473–84.
[18] Leong YS, Ker PJ, Hasnul MH, Khamis MA, Hannan MA, Jamaludin MZ, Looe HM, Portable device for transformer oil inhibitor content analysis using near-infrared spectroscopy wavelength. In: Proceedings of the IEEE-Industry-Applications-Society Annual Meeting, Electr Network, 2020 Oct 10–16, 2020.
[19] Trnka P, Hornak J, Prosr P, Michal O, Wang F. Various ageing processes in a paper-natural ester insulation system in the presence of copper and moisture. IEEE Access. 2010;8:61989–98.
[20] Testing and determination of specific additives in mineral insulating oils. 2015; TS EN 60666-2015.
[21] Chen W. IEC publication 590 (1977) Determination of aromatic hydrocarbon content in new mineral insulating oils. Power capacitors and reactive power compensation. 1978.
[22] Qian Y, Zhao Y, Wang Q. Application of infrared spectroscopy in monitoring the ageing process of transformer oil. In: Proceedings of the 2022 7th Asia Conference on Power and Electrical Engineering (ACPEE); 2022.
[23] Wang Y, Wang H, Qian Y, Wang Q. Study on the detection of acid value of insulating oil by infrared spectroscopy. Transformer. 2022;59:55–60.
[24] Liang S. Research on anti-ageing transformer oil and its effect on thermal ageing of insulating paper. Chongqing: Doctoral dissertation of Chongqing University; 2009.
[25] Yang T, Kou X, Zheng H, Chen S, Wang J, Hao J. Application of ternary hybrid insulating oil in old mineral insulating oil transformers at 35 kV in operation. Transformer. 2022;59:32–36.
[26] ASTM D1500-12:2018, Standard Test Method for ASTM Color of Petroleum Products (ASTM Color Scale), American Section of the International Association for Testing Materials.
[27] He L, Xiong C, Ma L, Li Z, Zhang X, Li H. Research on transformer life prediction method considering life differentiation phenomenon. Power Supply. 2022;39:93–100.
[28] IEC 60247:2002, Insulating liquids - Measurement of Relative Permittivity, Dielectric Dissipation Factor (tan d) and d.c. Resistivity.
[29] ISO 6295:1983, Petroleum Products - Mineral oils - Determination of Interfacial Tension of Oil Against Water - Ring Method.
[30] ASTM E1252:2021, Standard Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis.
[31] Peng H. Pearson correlation coefficient applied to medical signal correlation measurement. Electronics World. 2017;7:163.
[32] Polansky R, Prosr P, Vik R, Moravcova D, Pihera J. Comparison of the mineral oil lifetime estimates obtained by differential scanning calorimetry, infrared spectroscopy, and dielectric dissipation factor measurements. Thermochimica Acta. 2017;647:86–93.
[33] Calemma V, Iwanski P, Nali M, Scotti R, Montanari L. Structural characterization of asphaltenes of different origins. Energy Fuels. 1995;9:225–30.
[34] Castro LV, Vazquez F. Fractionation and characterization of Mexican crude oils. Energy Fuels. 2009;23:1603–9.
[35] Lievens C, Mourant D, He M, Gunawan R, Li X, Li C-Z. An FT-IR spectroscopic study of carbonyl functionalities in bio-oils. Fuel. 2011;90:3417–23.
[36] US-ASTM D02. 96. Standard Practice for Condition Monitoring of Used Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry: ASTM E2412-2010[S]. Philadelphia: ASTM; 2010.